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PATENT

No 1001555

Taking into account:

- d) The Greek Law 1733/87 on "Technology transfer, inventions, technological innovations and setting-up of an Atomic Energy Committee"
- e) The Ministerial Decision 15928/ΕΦΑ/1253 of the Greek Ministry of Industry, Energy and Technology on "Filing for an application for a Patent or Utility Model Patent before the Hellenic Industrial Property Organisation and record keeping"
- f) The application filed by the person concerned before the Hellenic Industrial Property Organisation on 31.05.1991 under No 910100234

We confer

This Patent accompanied by all relevant and attached documents certified according to the Greek Law (1733/87, Article 7, par. 11), to: 1) AGATZINI-LEONARDOU STYLIANI, residing at 76 Valtinon str., 114 74 Gizi, Athens, 2) DIMAKI DIMITRA, residing at 10 Manola str., 162 32 Vironas, Athens.

Entitled: "NICKEL AND COBALT RECOVERY FROM LOW-GRADE NICKEL OXIDE ORES BY THE METHOD OF HEAP LEACHING USING DILUTE SULPHURIC ACID AT AMBIENT TEMPERATURE."

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The General Manager

DIMITRIS KARLIS

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True translation of the attached document

S. Spanoudakis

NICKEL AND COBALT RECOVERY FROM LOW-GRADE NICKEL OXIDE ORES BY THE METHOD OF HEAP LEACHING USING DILUTE SULPHURIC ACID AT AMBIENT TEMPERATURE

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The present invention refers to a hydrometallurgical method for the extraction of nickel and cobalt from low to very low-grade nickel and cobalt oxide ores and specifically to the leaching (selective dissolution) of low to very low-grade nickel and cobalt oxide ores by dilute sulphuric acid at ambient temperature in order to extract nickel and cobalt from the ore in the form of an aqueous solution containing nickel and cobalt cations.

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The extraction of nickel from its oxide ores is industrially performed by pyrometallurgical (smelting to produce nickel matte and smelting to produce ferro-nickel), as well as hydrometallurgical (Caron's method by ammoniacal leaching and Freeport's Sulphur method by pressure leaching with sulphuric acid) methods. Nickel is extracted in Greece from its oxide ores as ferro-nickel by the application of a pyrometallurgical method.

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However, most of these methods are energy consuming and were applied when oil was cheap and was believed that it would constitute a cheap fuel for a long period of time yet. Hence, all pyrometallurgical methods as well as Caron's hydrometallurgical method, due to drying and reductive roasting of the ore, require a high energy consumption (500-1000 MJ per kg of nickel at the final product). The Freeport's sulphur hydrometallurgical method removed the energy consuming stage of the thermal treatment of the ore, but again, it cannot economically be used for low-grade Ni ore, since it requires expensive equipment (autoclaves, flash tanks), high leaching temperatures (230°C-260°C) and presents serious technical problems because of its corrosive nature^(1,2,3).

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For these reasons, none of the above methods can economically be applied for the nickel oxide ores with a nickel content less than 1%.

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But, if one considers the following:

- a. the nickel content of the nickel oxide ores, that are now being exploited in Greece, is between 0.95% and 1%, which is a limiting grade at the present economic conditions of the metal market, which are considered to be especially favourable for nickel,

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b. the proved reserves of nickel oxide ores in Greece with a nickel content above 0.95% are estimated to only 25×10^6 t, while those with nickel between 0.95% and 0.75% are estimated to more than 200×10^6 t,

- 5 c. the international statistics foresee a fall of nickel price within the next five years⁽⁵⁾ and, hence, an increase of the nickel cut-off grade of the deposits that are exploitable,

it becomes obvious that for the exploitation of the low-grade nickel and cobalt oxide ores, both internationally and particularly in Greece, a new/new method/methods with a much lower energy
10 cost than that of the existing methods is/are required.

The present invention, as described below, solves economically the problem of nickel and cobalt extraction, in the form of an aqueous solution, from low to very low nickel oxide ores. The invention consists in finding all the factors and determining the values of these factors, as well
15 as the development of the suitable pretreatment of the ore, so that, by using -for the first time at an international level- the well known method of "heap-leaching" on low-grade nickel oxide ores, the nickel and cobalt extraction becomes possible, at a large extend, using a dilute solution of sulphuric acid as the leaching agent.

20 The amenability of nickel oxide ores to heap leaching, using sulphuric acid was simulated, at the laboratory scale, by column leaching, after having resolved all technical problems that arose from the nature of the ore. These problems are the following:

a) The ore contains clay components which create a big problem as far as the percolation of the leach solution (lixiviant) through the ore column is concerned since the swelling of the clay
25 minerals reduces the established porosity. It was found that this problem is solved if, before the placement of the ore into a column or in heaps, the ore acquires an approximately 10% moisture. This can be accomplished by wetting the ore with either water or leach solution (lixiviant). Hence, during stacking of the ore, the clay constituents have already swelled, while any existing fines are agglomerated, with the result that the porosity is modulated only by the
30 weight of the material and by the conditions the ore is being stacked.

b) The determination of the particle size and size distribution of the ore, that is placed into a column or in a heap, constitutes a second important problem as the small particle size on the one hand favours leaching, because it leads to a grater surface area and contact surface
35 between the ore and the leach solution (lixiviant), making the extraction more efficient while, on the other hand, it impedes the percolation of the leach solution through the column or heap, due to low porosity, with the consequence that the column or heap is often blocked. Various ore particle sizes were tried and it was found that ores with particles even as large as 3cm can

be used, without creating any problems to the percolation of the leach solution (lixiviant), while, at the same time, the efficiency of the extraction is very good. This fact is important since the very expensive grinding of the material is avoided, thus making the suggested method more economic.

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After having tested, two techniques of application (I and II) of the heap leaching method are suggested. The techniques are described below as far as their general characteristics are concerned. The details of their application are determined based on the special characteristics of the ore and of the method that is decided to be employed for the recovery of nickel from the leach liquor (pregnant solution).

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TECHNIQUE I

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The leach solution is recycled inside the column as many times as it is required so that no increase of the nickel concentration is measured any more. It is then replaced by a fresh solution with which the previous process is repeated etc. The thus obtained final metal containing solutions (leach liquors or pregnant solutions) have low nickel concentrations, in general. Their composition depends on the type of the ore and the stage of leaching.

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In applying the above technique of the heap leaching method, six different experiments were carried out in fiberglass columns, 10cm in diameter. Each column was loaded with nickel oxide ore from a deposit found in Greece and had the chemical composition shown in Table 1 (sample A).

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For the column leaching, the value of 4.5 l/day was selected as the leach solution flowrate, though it was possible to use higher flowrates. In cases of application of the method, a flowrate ranging from 100 l/day/m² to 800 l/day/m² is recommended, with a preference towards the intermediate values of 300-600 l/day/m².

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The columns differed only with respect to the ore heights inside them and, consequently, only in the amount of the material being leached. Hence, apart from the study of the factor of the column height, sufficient data were collected in order to be able to anticipate the behavior of the system on larger experimental scales.

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TABLE 1Chemical analysis of the nickel oxide ores

Component	Percentage (%)	
	<u>SAMPLE A</u>	<u>SAMPLE B</u>
Ni	0.73	0.73
Co	0.047	0.056
Fe ₂ O ₃	18.11	26.90
Cr ₂ O ₃	2.19	1.75
Al ₂ O ₃	6.70	7.10
MgO	2.30	1.45
CaO	5.68	8.40
SiO ₂	45.34	41.95
Ignition loss	11.00	

The particular operational characteristics of each column are shown in Table 2. The initial sulphuric acid concentration in the leach solution (lixiviant) was chosen to be 1N. It was found that nickel extraction occurs as long as the pH of the leach solution is lower than 1-1.3. At pH values higher than the above range, nickel extraction is negligible. By using this heap leaching technique, the free acid remaining in the leach liquor is not exploited.

TABLE 2Operational characteristics of the laboratory leaching columns

Column height (m)	Height used (m)	Ore weight (kg)
2.50	2.20	22
2.10	1.80	18
1.80	1.50	15
1.50	1.20	12
1.20	1.00	10
1.00	0.80	8

The results obtained are given in Figures 1-13, from where the following conclusions are derived:

1. The iron and nickel extraction rates differ significantly. Thus, while the Fe/Ni ratios in leach liquors (pregnant solutions) resulting from agitation leaching, at atmospheric pressure, of nickel oxide ores (cited in the Greek and foreign bibliography⁽⁶⁾) are generally high, greater than 5, by using the suggested technique the Fe/Ni ratio obtained ranges from 1/1 to 2/1, for nickel recoveries up to approximately 70%. Beyond this nickel percentage recovery, an intense iron dissolution occurs while that of nickel is very slow.
2. The nickel and iron extraction, together with the extraction of other elements, starts after the extraction of any calcium found is completed. The extracted calcium is then precipitated as gypsum, during successive recyclings. Hence, after three to four recyclings, the solution does not contain any calcium.
3. By increasing the weight of the ore that is leached in the column, the acid consumption as well as the number of the required recyclings are reduced while the time required to achieve a certain nickel extraction increases.

The leach liquors (pregnant solutions) that are produced by the application of the above heap leaching technique are, in general, poor and their exact composition depends on the type of the ore and the stage the leaching has reached. A characteristic composition of the leach liquor resulting from the column leaching of the used ore is given in Table 3.

According to another application of the Technique I to a sample of a nickel and cobalt oxide ore, originating from a different deposit in Greece (Sample B) with a chemical composition given in Table 1, particles size -3 mesh and applied leach solution flowrate equal to 2.1 l/day, the final nickel content of the leach liquor was higher (1.8g/l) and equal to that of iron. This ore had much better leaching characteristics.

TABLE 3

Typical chemical analysis of a leach liquor produced by Technique I

Ni	1.1.5 g/l
Fe	1.5-3 g/l
Co	as 0.07 g/l
Mg	1.5-2 g/l
Cr	0.2-0.3 g/l
Al	1-2-2 g/l
Ca	10-20ppm

TECHNIQUE II

The leach solution (lixiviant) is recycled through the ore in the column after pH adjustment to a predetermined value, thus performing a number of leach cycles. The leaching -pH adjustment process is considered to have been completed when nickel concentration in the leach liquor (pregnant solution) stops to increase or when it is so judged. The thus resulting leach liquor can subsequently be used, by exactly the same way, to leach ore in another column, until the liquor acquires the desired nickel concentration. It should be stressed here that this continuous recycling of the leach liquor and therefore, the enrichment of the leach liquor in metal ions, does not suppress leaching, but only if the solution gets saturated with respect to an element, which is something that leads to its precipitation in the form of a chemical precipitate.

The application of this technique diminishes water requirements while, at the same time, it allows the utilization of the free sulphuric acid remaining in the final leach liquor.

According to an application of this technique of heap leaching, two experiments were performed using an ore with a chemical analysis given in Table 1 (Sample A). The leach solution flowrate was fixed to 4.5 l/day, the initial height of each ore column at 1.20m, while the acidity of the solution was 1N (pH about 0.5) for the first column and 2N (pH about 0.3) for the second column.

According to another application of the same technique, the lowest possible acidity, that the leach solution must have, was studied. It was found that the lower acidity limit was 0.25M, corresponding to pH of about 1.2, that is to say a value equal to the one that was found to be also marginal for the leaching experiments by Technique I. This fact confirms that cessation of nickel extraction at pH values higher than 1.2 is exclusively due to the reduced acidity of the solution rather than to the increased nickel concentration. Therefore, provided that the acidity is the right one ($>0.25M$), nickel concentration in the solution can increase until the leach liquor becomes saturated.

According to another application of Technique II, the effect of the ore particle size (-3mesh, -3cm) as well as that of various size fractions of the same ore (-3cm, +3mesh, -3mesh) were studied.

The results are given in Figures 14-25, giving rise to the following conclusions:

- i) The Fe/Ni ratio in the leach liquor does not exceed 5/1, even for nickel recoveries higher than 75%.

ii) The acid consumption is less by using Technique II. The observed values are given in Figures 14 and 13, from where it can be seen that the acid consumption is reduced as the quantity of the ore being leached increases, while it increases as the initial sulphuric acid concentration in the leach solution is reduced. Given that a significant percentage (up to 10%) of the acid consumption is due to the presence of calcium in the ore, the possibility to reduce the sulphuric acid consumption by the use of a carefully extracted, and therefore with a small gangue material (limestone) content, ore is obvious.

iii) Shorter extraction times are achieved by this technique.

iv) No significant differences in extraction rates and final nickel and cobalt recoveries were observed, for any of the size fractions and particle ore sizes that were tested.

Apart from the above conclusions, during the experimental application of the heap leaching method to nickel oxide ores and irrespective of the applied technique (I or II), the following were found out and are suggested by the inventors for the commercial application of the method:

i) The increase in acidity significantly shortens leaching time.

ii) The ratio of the heap ore weight to the total leach solution (lixiviant) volume selected plays an important role regarding the final chemical composition of the leach liquor. The value of this ratio is chosen on the basis of the water deposits, the weather conditions, the acid concentration used and the desired leaching time.

iii) The final nickel concentration of the leach liquor depends on the aluminium concentration in the same solution. Aluminium concentrations higher than 11-12g/l induce nickel precipitation, thus reducing final nickel recovery. Therefore, depending on the leachability of the aluminium in the ore, it is recommended that the ratios of the ore weight to leach solution volume do not exceed 2.5/1.

iv) It was found that washing the heaps with water is only required after completion of leaching while it does not affect leaching if it takes place at an intermediate stage.

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CLAIMS

1. The suggested method of leaching nickel and cobalt oxide ores in heaps is characterized by that it applies, for the first time, the heap leaching method for the extraction of nickel and cobalt from nickel and cobalt oxide ores, using multiple recycling of dilute sulphuric acid solution through one or more ore heaps. The pH of the solution may or may not be adjusted to a predetermined value, prior to recycling, depending on the leaching technique applied (I or II).
2. The method of heap leaching of nickel and cobalt oxide ores, according to claim 1, is characterized by that it accomplishes extraction of nickel and cobalt from high-grade ores and, also, from low-grade ores, that cannot be treated economically by existing methods, by using dilute sulphuric acid solutions and achieving consumptions that are much lower than those which characterize other methods of leaching nickel and cobalt oxide ores with sulphuric acid.
3. The method of heap leaching of nickel and cobalt oxide ores, according to claims 1 and 2, is characterized by that it significantly improves the nickel recovery and the consumptions of sulphuric acid and water if it is combined with multiple recycling of the same solution, into the same or different ore heaps, after previous adjustment of the solution pH to a predetermined value.
4. The application of the suggested method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that it requires even wetting of the ore, prior to its placement on the leach pad, with so much water or leach solution (lixiviant) as to raise the normal moisture content of the ore by approximately 10%.
5. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that the sulphuric acid concentration in the leach solution (lixiviant) must be at least 0.25M (0.5N). By increasing the acidity of the solution, the extraction time (leaching time) is reduced but the Fe/Ni ratio in the leach liquor (pregnant solution) increases.
6. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that the ratio "ore weight / leach solution volume" is recommended to be kept within the range 5/4-2/1 for a more efficient extraction.
7. The suggested method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that it can be applied to nickel and cobalt oxide ores with a minimum nickel content of 0.3%.
8. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that the nickel recovery and the extraction time are not affected by the ore particle size, provided this is less than 3cm.

9. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that the extraction of metals contained in the ore obey the following rules:

5 a) The extractions of nickel, iron, cobalt, magnesium, chromium and aluminium take place after the calcium extraction, which can occur at very low sulphuric acid concentrations (lower than 0.25M) in the leach solution, has been completed. In the case there is no calcium in the ore, the leaching of the above metal cations start directly and simultaneously.

10 b) After the extraction (leaching) of the various metal cations, that are present in the ore, has started, calcium is gradually being precipitated as gypsum, within the heap, due to an increase of the sulphate anions concentration in the leach liquor.

10. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that the rate of nickel extraction is constant and much higher than the rates of iron and chromium, while it is of the same order of magnitude with the extraction
15 rates of magnesium and cobalt, until nickel recovery reaches 70-72%. Beyond this range, the nickel extraction rate is significantly reduced while that of iron increases.

SUMMARY

NICKEL AND COBALT RECOVERY FROM LOW-GRADE NICKEL OXIDE ORES BY
THE METHOD OF HEAP LEACHING USING DILUTE SULPHURIC ACID AT AMBIENT
5 TEMPERATURE.

A new hydrometallurgical method for nickel and cobalt extraction from low to very low-grade
nickel and cobalt oxide ores is suggested, using heap leaching of the ore with sulphuric acid
solution.

10 The recovery of nickel and cobalt from low or high grade nickel and cobalt oxide ores is made
possible by the suggested method, whereby dilute sulphuric acid solution is recycled a number
of times through one or more ore heaps. According to the suggested techniques of application,
the acidity of the sulphuric acid solution is adjusted to a predetermined value before each
15 recycling (Technique II) or the sulphuric acid solution is replaced by a new one when its acidity
falls below a predetermined value (Technique I). The extractability of the various metal cations
present in the ore is given in Figures 1-25. A study of various factors that might affect the ore
leaching was also conducted and the optimum leaching conditions, based on the obtained
experimental data, were determined.

FIGURE CAPTIONS

- Figure 1. Percent extraction of Ni, Fe, Mg, Co, Cr, Al as a function of time, using the solution replacement technique I
- Figure 2. Percent extraction of Ni, Fe, Mg, Co, Cr, Al as a function of time, using the solution replacement technique I
- Figure 3. Percent extraction of Ni, Fe, Mg, Co, Cr, Al as a function of time, using the solution replacement technique I
- Figure 4. Percent extraction of Ni, Fe, Mg, Co, Cr, Al as a function of time, using the solution replacement technique I
- Figure 5. Percent extraction of Ni, Fe, Mg, Co, Cr, Al as a function of time, using the solution replacement technique I
- Figure 6. Percent extraction of Ni, Fe, Mg, Co, Cr, Al as a function of time, using the solution replacement technique I
- Figure 7. Percent extraction of Ni as a function of time for various column heights, using the solution replacement technique I
- Figure 8. Percent extraction of Ni as a function of the leaching cycles for various column heights, using the solution replacement technique I
- Figure 9. Percent extraction of Fe as a function of time for various column heights, using the solution replacement technique I
- Figure 10. Percent extraction of Fe as a function of the leaching cycles for various column heights, using the solution replacement technique I
- Figure 11. Percent extraction of Co as a function of time for various column heights, using the solution replacement technique I
- Figure 12. Percent extraction of Co as a function of the number of leaching cycles for various column heights, using the solution replacement technique I
- Figure 13. Relative acid consumption for various column heights, using the solution replacement technique I
- Figure 14. Relative acid consumption for various sulphuric acid concentrations in the leach solution
- Figure 15. Percent extraction of Ni, Fe, Mg, Co, Cr, Al as a function of time, using the pH adjustment technique II
- Figure 16. Percent extraction of Ni, Fe, Mg, Co, Cr, Al as a function of time, using the pH adjustment technique II
- Figure 17. Percent extraction of Ni as a function of the acidity of the leach solution
- Figure 18. Percent extraction of Fe as a function of the acidity of the leach solution

Figure 19. Percent extraction of Co as a function of the acidity of the leach solution

Figure 20. Percent extraction of Ni for various particle sizes and size fractions

Figure 21. Percent extraction of Fe for various particle sizes and size fractions

Figure 22. Percent extraction of Co for various particle sizes and size fractions

Figure 23. Percent extraction of Mg for various particle sizes and size fractions

Figure 24. Percent extraction of Cr for various particle sizes and size fractions

Figure 25. Percent extraction of Al for various particle sizes and size fractions

CLAIMS

1.The suggested method of leaching nickel and cobalt oxide ores in heaps is characterized by that it applies, for the first time, the heap leaching method for the extraction of nickel and cobalt from nickel and cobalt oxide ores, using multiple recycling of dilute sulphuric acid solution through one or more ore heaps. The pH of the solution may or may not be adjusted to a predetermined value, prior to recycling, depending on the leaching technique applied(I or II).

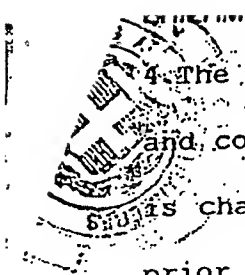
2.The method of heap leaching of nickel and cobalt oxide ores, according to claim 1, is characterized by that it accomplishes extraction of nickel and cobalt from high-grade ores and, also, from low-grade ores, that cannot be treated economically by existing methods, by using dilute sulphuric acid solutions and achieving consumptions that are much lower than those which characterize othe methods of leaching nickel and cobalt oxide ores with sulphuric acid.

3.The method of heap leaching of nickel and cobalt oxide ores, according to claims 1 and 2, is characterized by that it significantly improves the nickel recovery and the consumptions of sulphuric acid and water if it is combined with multiple recycling of the same solution, into the same or different ore heaps, after previous adjustment of the solution pH to a predetermined value.

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4. The application of the suggested method of leaching nickel and cobalt oxide ores in heaps, according to claims 1,2 and 3, is characterized by that it requires even wetting of the ore, prior to its placement on the leach pad, with so much water or leach solution (lixiviant) as to raise the normal moisture content of the ore by approximately 10%.

5. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1,2 and 3, is characterized by that the sulphuric acid concentration in the leach solution (lixiviant) must be at least 0,25M(0.5N). by increasing the acidity of the solution, the extraction time (leaching time) is reduced but the Fe/Ni ratio in the leach liquor (pregnant solution) increases.

6. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1,2 and 3, is characterized by that the ratio "ore weight/leach solution volume" is recommended to be kept within the range 5/4-2/1 for a more efficient extraction.


7. The suggested method of leaching nickel and cobalt oxide ores in heaps, according to claims 1,2 and 3, is characterized by that it can be applied to nickel and cobalt oxide ores with a minimum nickel content of 0.3%

8. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1,2 and 3, is characterized by that

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the nickel recovery and the extraction time are not affected by the ore particle size, provided this is less than 3cm.

9. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that the extraction of metals contained in the ore obey the following rules:

a) The extractions of nickel, iron, cobalt, magnesium, chromium and aluminium take place after the calcium extraction, which can occur at very low sulphuric acid concentrations (lower than 0,25M) in the leach solution, has been completed. In the case there is no calcium in the ore, the leaching of the above metal cations start directly and simultaneously.

b) After the extraction (leaching) of the various metal cations, that are present in the ore, has started, calcium is gradually being precipitated as gypsum, within the heap, due to an increase of the sulphate anions concentration in the leach liquor.

10. The method of leaching nickel and cobalt oxide ores in heaps, according to claims 1, 2 and 3, is characterized by that the rate of nickel extraction is constant and much higher than the rates of iron and chromium, while it is of the same order of magnitude with the extraction rates of magnesium and cobalt, until nickel recovery reaches 70-72%. Beyond this range, the nickel extraction rate is significantly reduced while that of iron increases.

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SUMMARY

NICKEL AND COBALT RECOVERY FROM LOW-GRADE NICKEL OXIDE ORES BY
THE METHOD OF HEAP LEACHING USING DILUTE SULPHURIC ACID AT
AMBIENT TEMPERATURE

A new hydrometallurgical method for nickel and cobalt extraction from low to very low-grade nickel and cobalt oxide ores is suggested, using heap leaching of the ore with sulphuric acid solution.

The recovery of nickel and cobalt from low or high grade nickel and cobalt oxide ores is made possible by the suggested method, whereby dilute sulphuric acid solution is recycled a number of times through one or more ore heaps. According to the suggested techniques of application, the acidity of the sulphuric acid solution is adjusted to a predetermined value before each recycling (Technique II) or the sulphuric acid solution is replaced by a new one when its acidity falls below a predetermined value (Technique I). The extractability of the various metal cations present in the ore is given in Figures 1-25. A study of various factors that might affect the one leaching was also conducted and the optimum leaching conditions, based on the obtained experimental data, were determined.

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